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# Strength and Fracture Toughness of Ultrafine Grained Alumina Ceramic

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**Colorado Springs, Colorado, USA**

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14. ABSTRACT

15. SUBJECT TERMS

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# Outline

- Introduction
- Experimental Procedures
- Results and Discussion
  - Bending Process
  - Fracture Morphology
  - Statistical Analysis
  - Fracture Toughness
- Conclusions
- Acknowledgements

# Introduction

- **Griffith's Theory**

$$\sigma = \frac{K_{Ic}}{Y\sqrt{a}}$$

σ -- Flexure strength

$K_{IC}$ -- Fracture toughness

*Y* -- Stress intensity factor

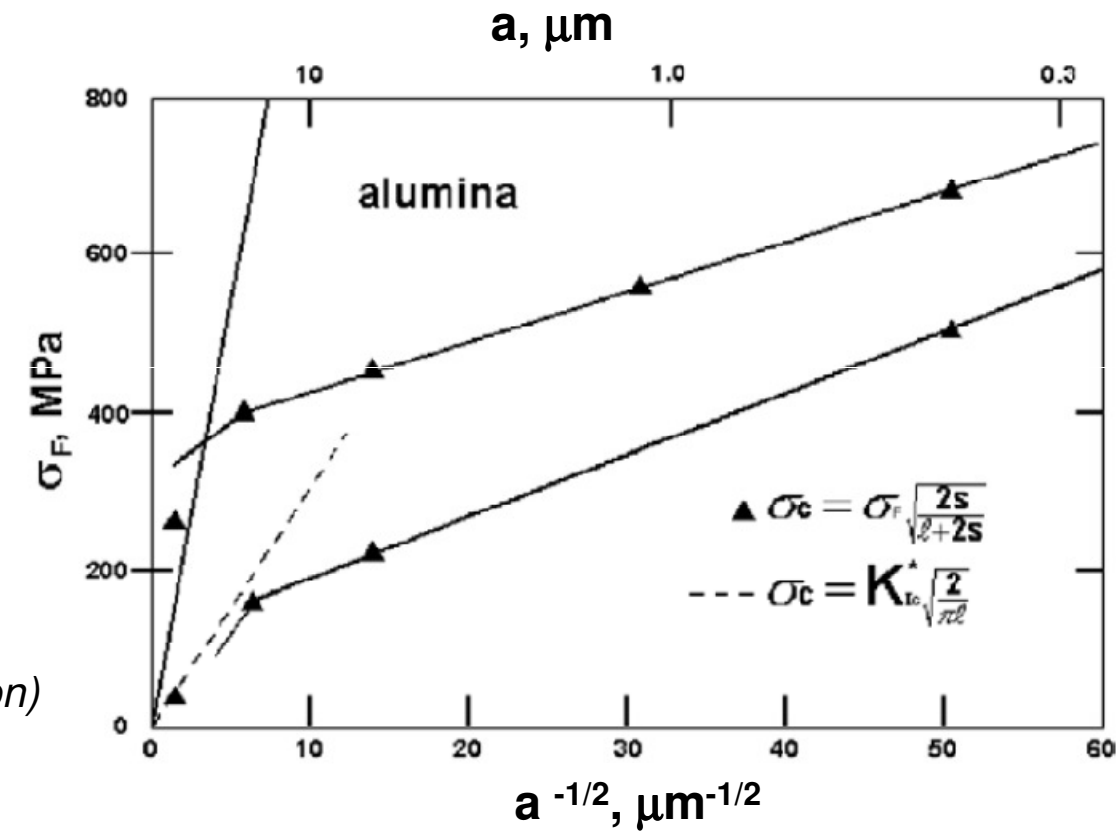
*a* -- Critical flaw size

$$\sigma = \sqrt{\frac{2E\gamma}{\pi a}}$$ *(Modified version)*

*E* -- Elastic modulus

$\gamma$ -- Surface energy

*a* -- Critical flaw size



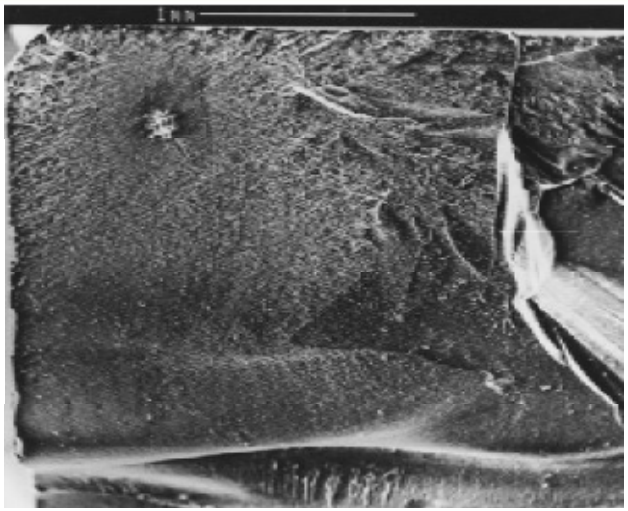

**Bending strength dependence on grain size of alumina**

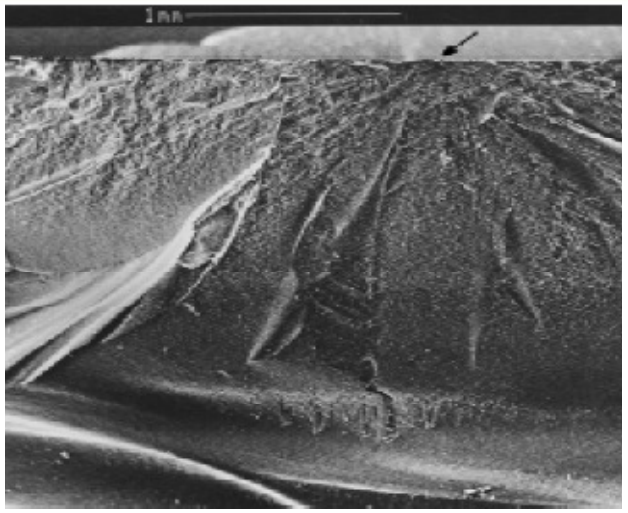


**R.W. Armstrong, *Inter. J. Refract. Metal. Hard Mater*, 19, 2001, 251-255.**

# Introduction

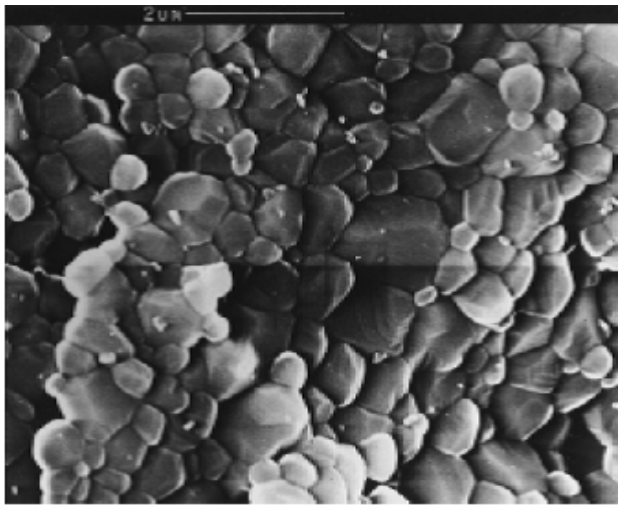
**Volume Flaw**

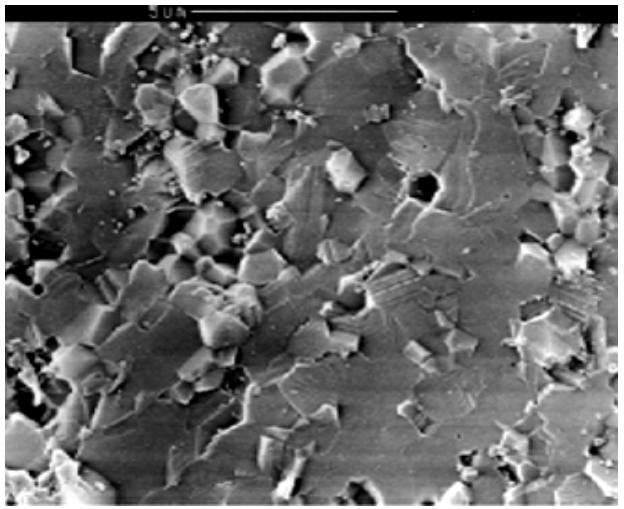



**Surface Flaw**

**Intergranular Fracture**



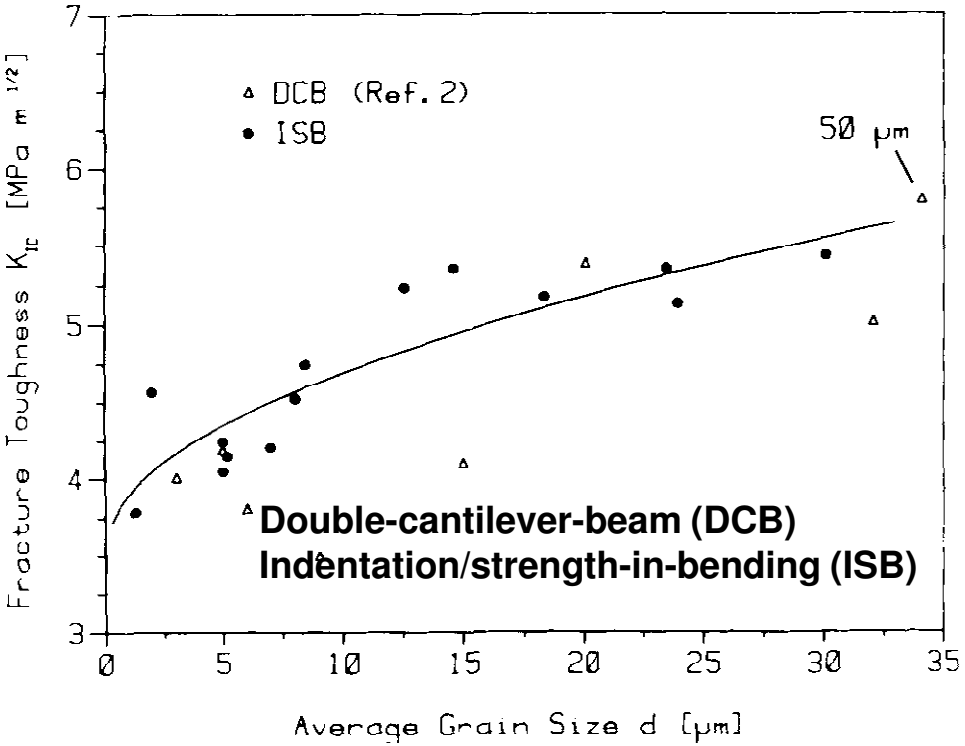

**Transgranular Fracture as well as Intergranular Fracture**

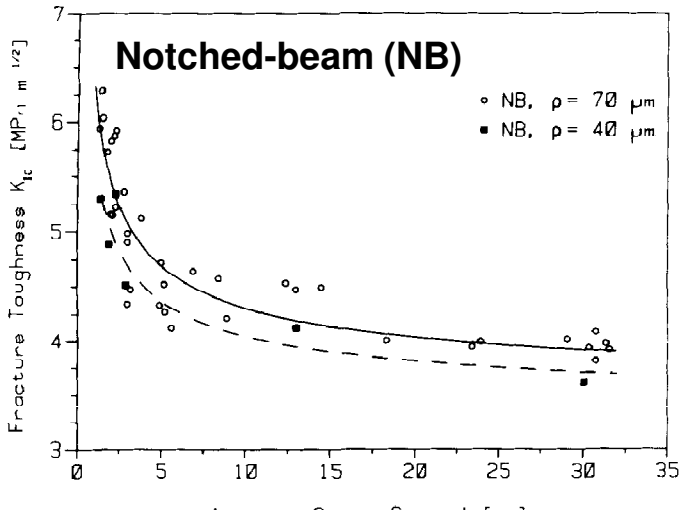


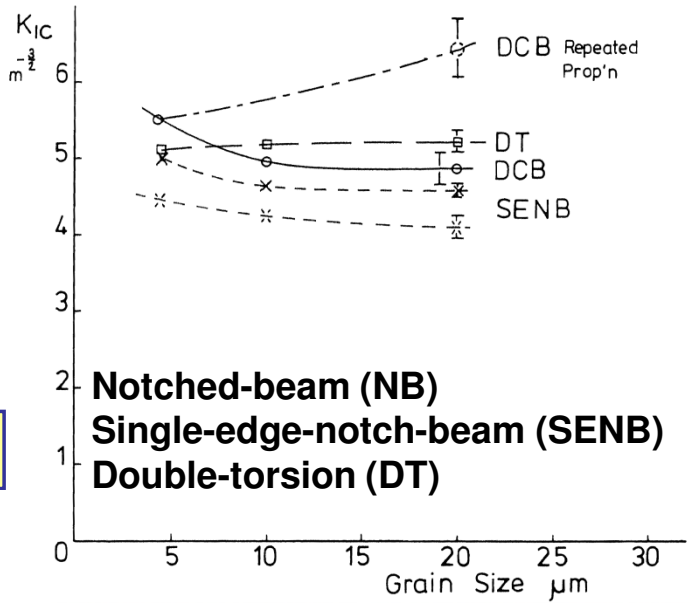
A. Krell, *J. Am. Ceram. Soc.*, 81[7], 1998, 1900-1906.

# Introduction

- **Dependence of fracture toughness of alumina on grain size**



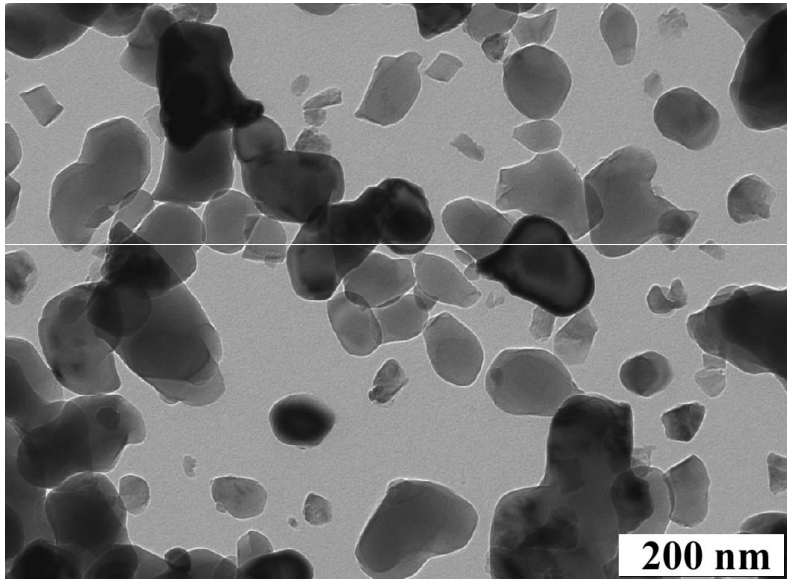

**Previous work on ultrafine grained alumina is limited.**


B. Mussler et al., *J. Am. Ceram. Soc.*, 1982, 65(11), 566-572

P. L. Pratt, *Fracture*, 1977, 3, 909-912

# Experimental Procedures

**$\alpha$-$Al_2O_3$ (TM DAR 6914)**
**~100nm, 99.99 %**

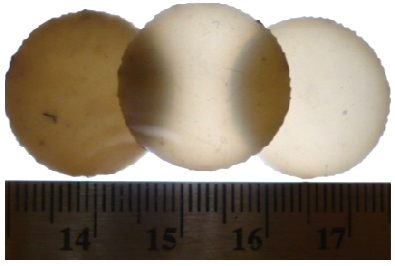



**Composition of feedstock powder**

| Powder Composition | $\alpha$ - $Al_2O_3$ (wt. %) | Impurity (ppm) | | | | | |
|---|---|---|---|---|---|---|---|
| | 99.99 | Si | Fe | Na | K | Ca | Mg |
| | | 1 | 8 | 3 | 2 | 3 | 1 |

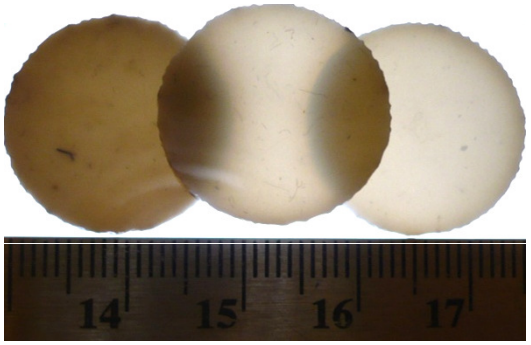
**Spark Plasma Sintering**
**(SPS-825S, DR. Sinter)**

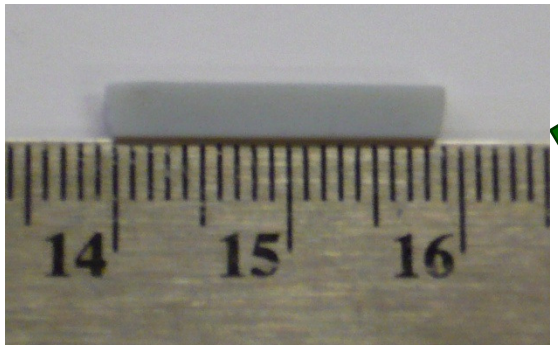
**As-Sintered Samples**

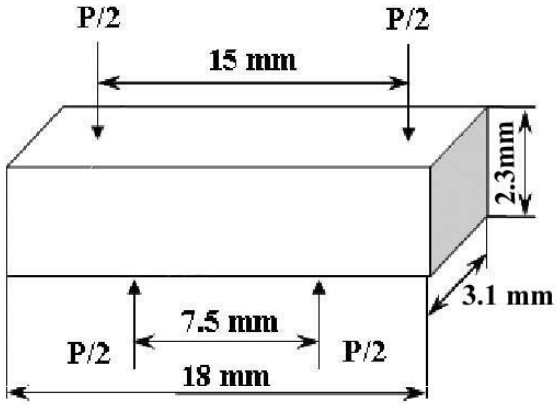


# Experimental Procedures

**As-Sintered Samples**

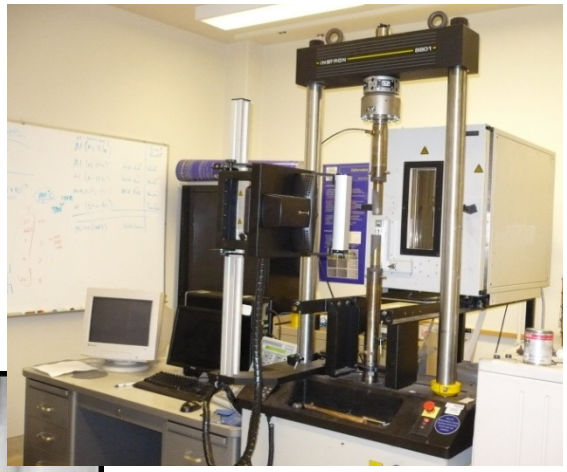


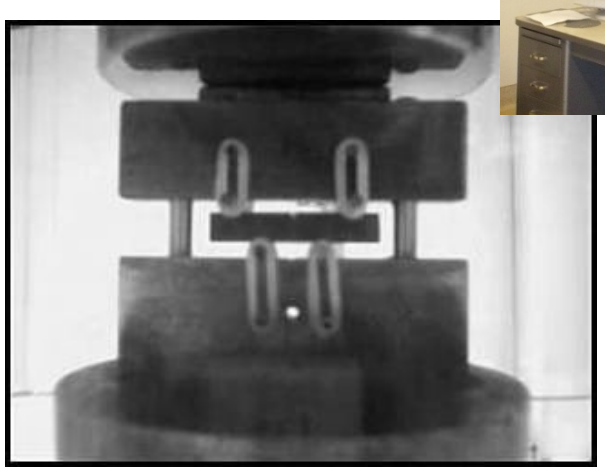




**Dimensions of bending beam**

**Instron universal testing machine**



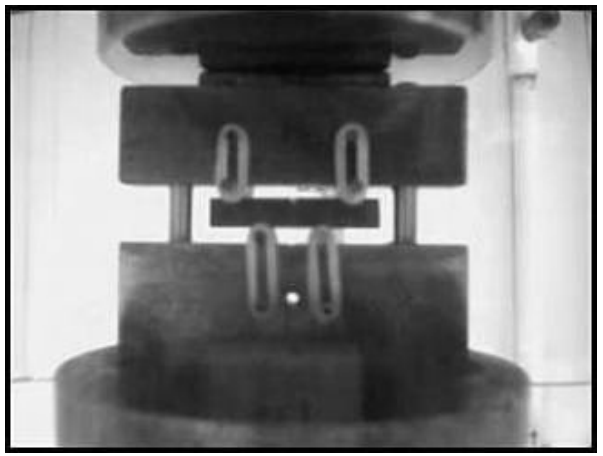
**Strain rate: 0.0015 $s^{-1}$**



**miniature 4-point bend test fixture**

# Experimental Procedures

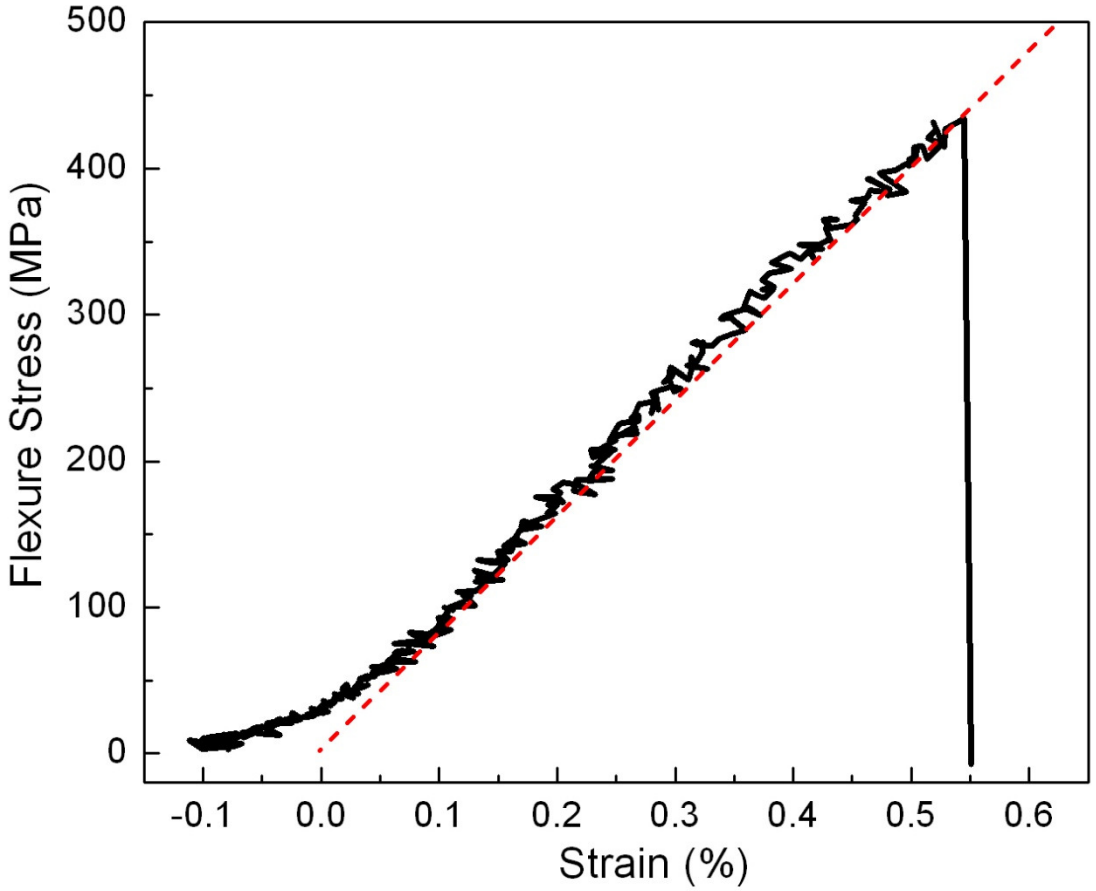
- Determination of Fracture Toughness of Ultrafine Grained Alumina
  - Vickers indentation
  - Surface Crack in Flexure
  - Single-edge-V-notched beam (SEVNB)



# Results and Discussion

- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness

# Results and Discussion



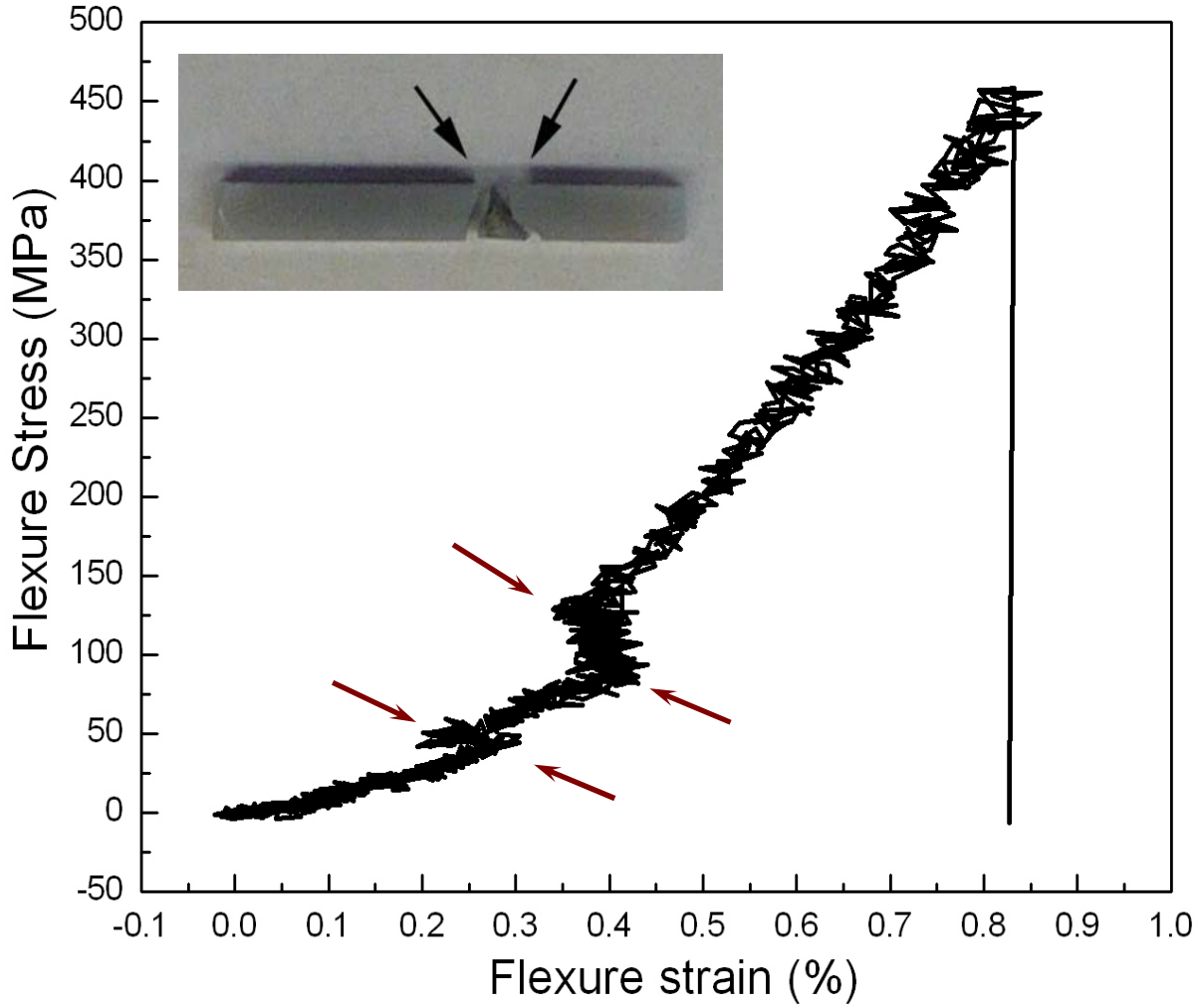

**Typical Bending Stress-Strain Curve**

- **Average Grain Size -- 363 40 nm**
- **Relative Density -- 99.1%**
- **Strength -- 434MPa**

- **Sample preparation methods (chamfering the edges; annealing samples for a short time before bending)**
- **Preloading conditions and strain rate**

*** Strain was calculated from gauge length (Toe compensation).**

# Results and Discussion



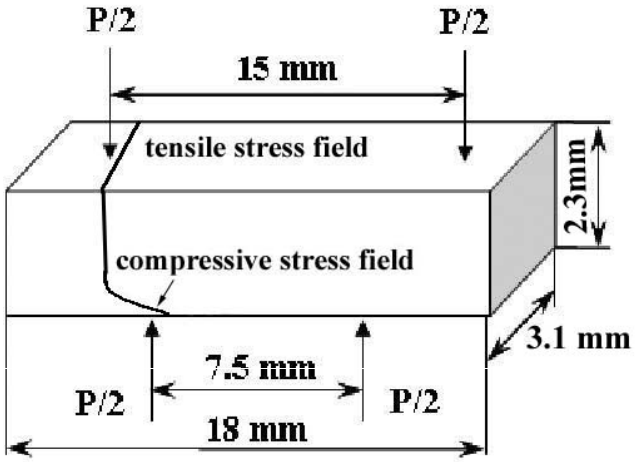

- **Average Grain Size -- 320 20 nm**
- **Relative Density -- 99.5%**
- **Strength -- 458MPa**

*** Strain was calculated from gauge length.**

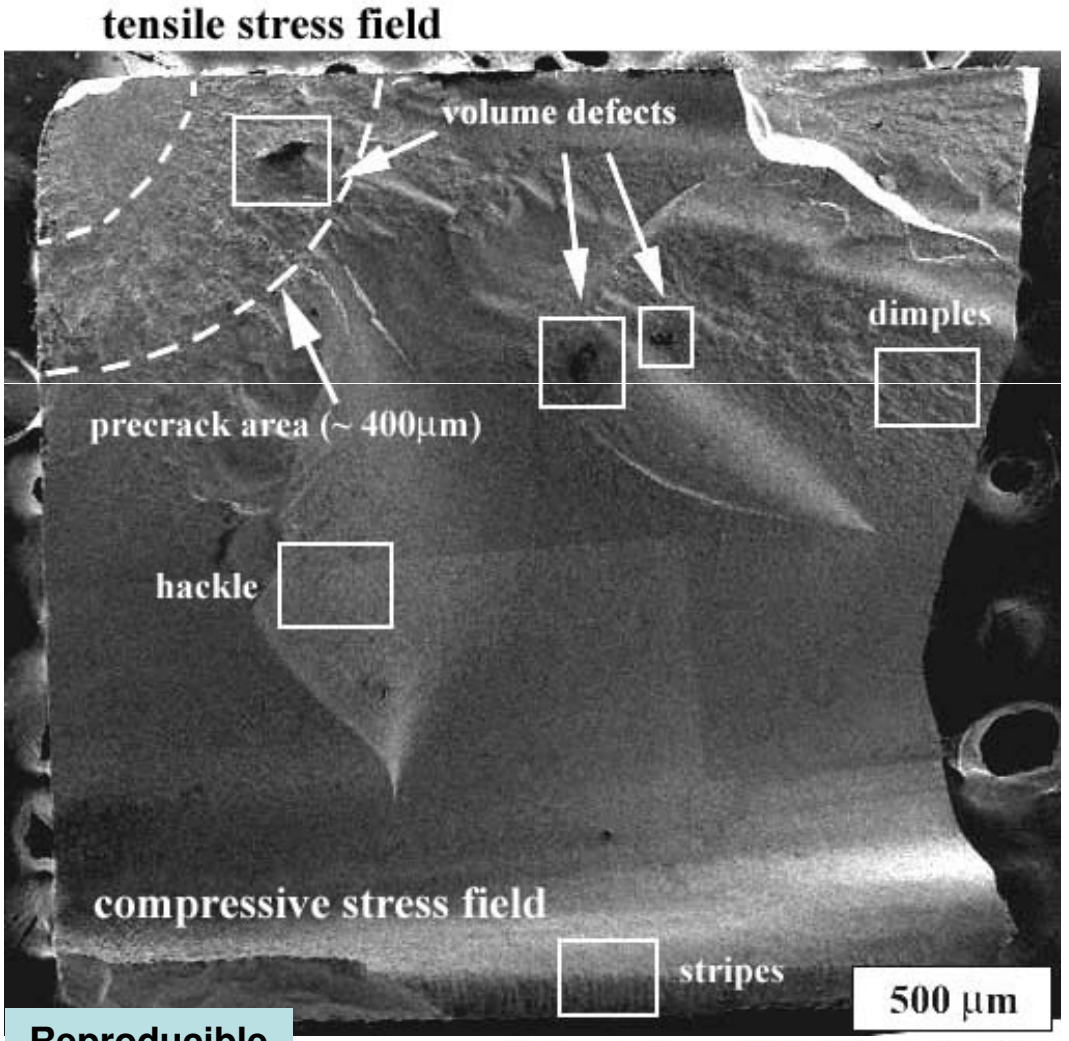
# Results and Discussion

- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness

# Results and Discussion

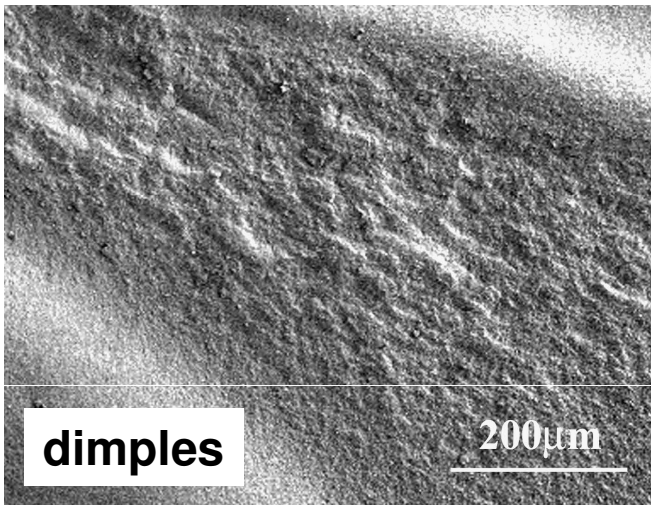


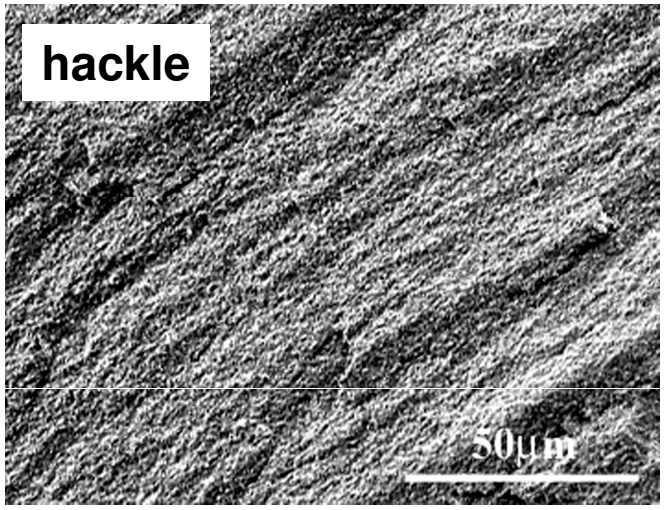

- **Average Grain Size -- 363 40 nm**
- **Relative Density -- 99.1%**
- **Strength -- 434MPa**

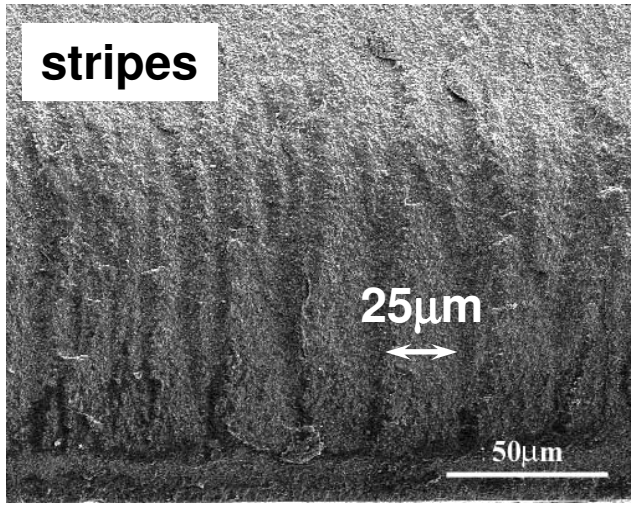



**Reproducible**

# Results and Discussion



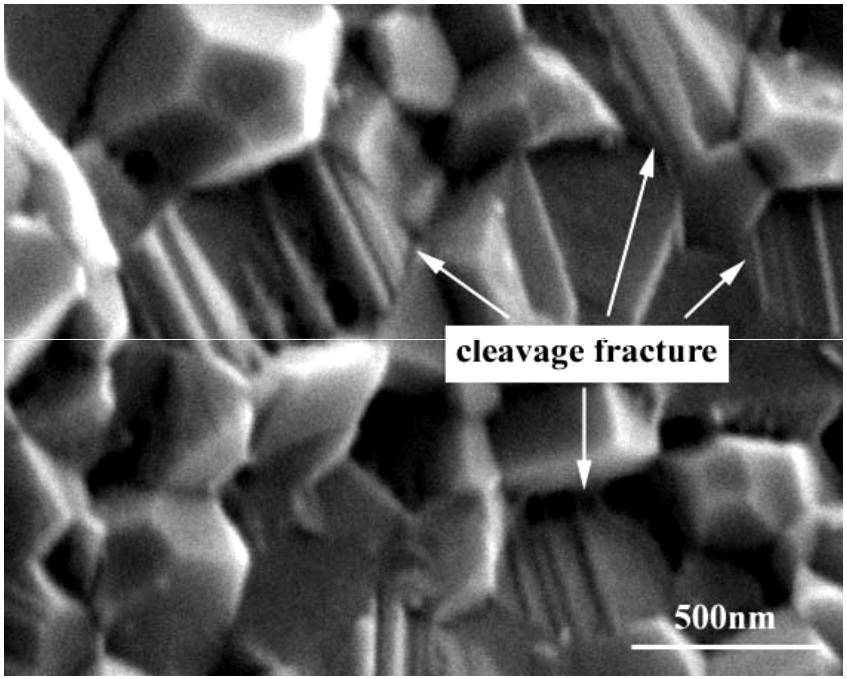



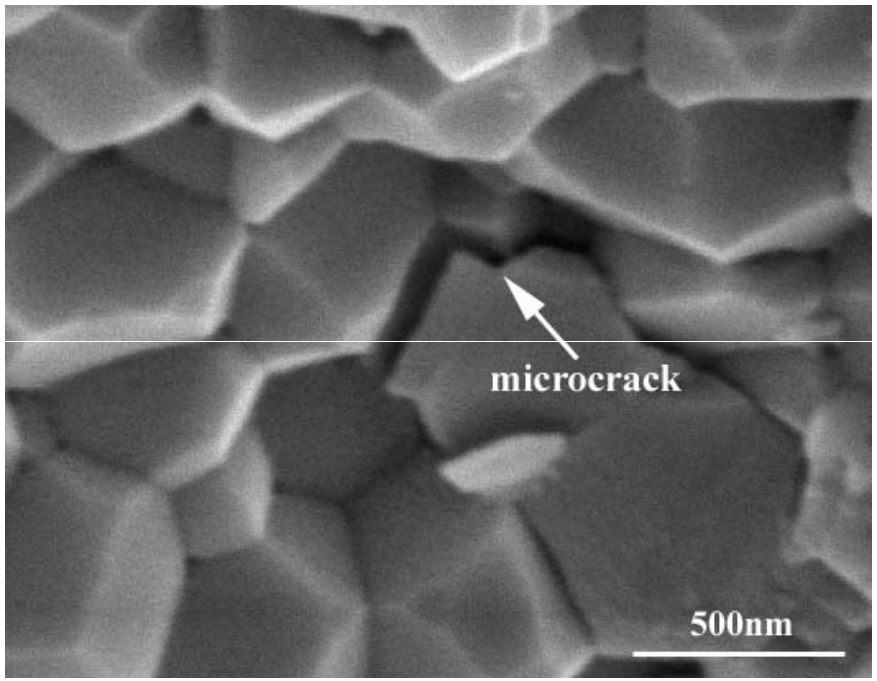

Local magnifications

- Average Grain Size -- 363 40 nm
- Relative Density -- 99.1%
- Strength -- 434MPa

# Results and Discussion



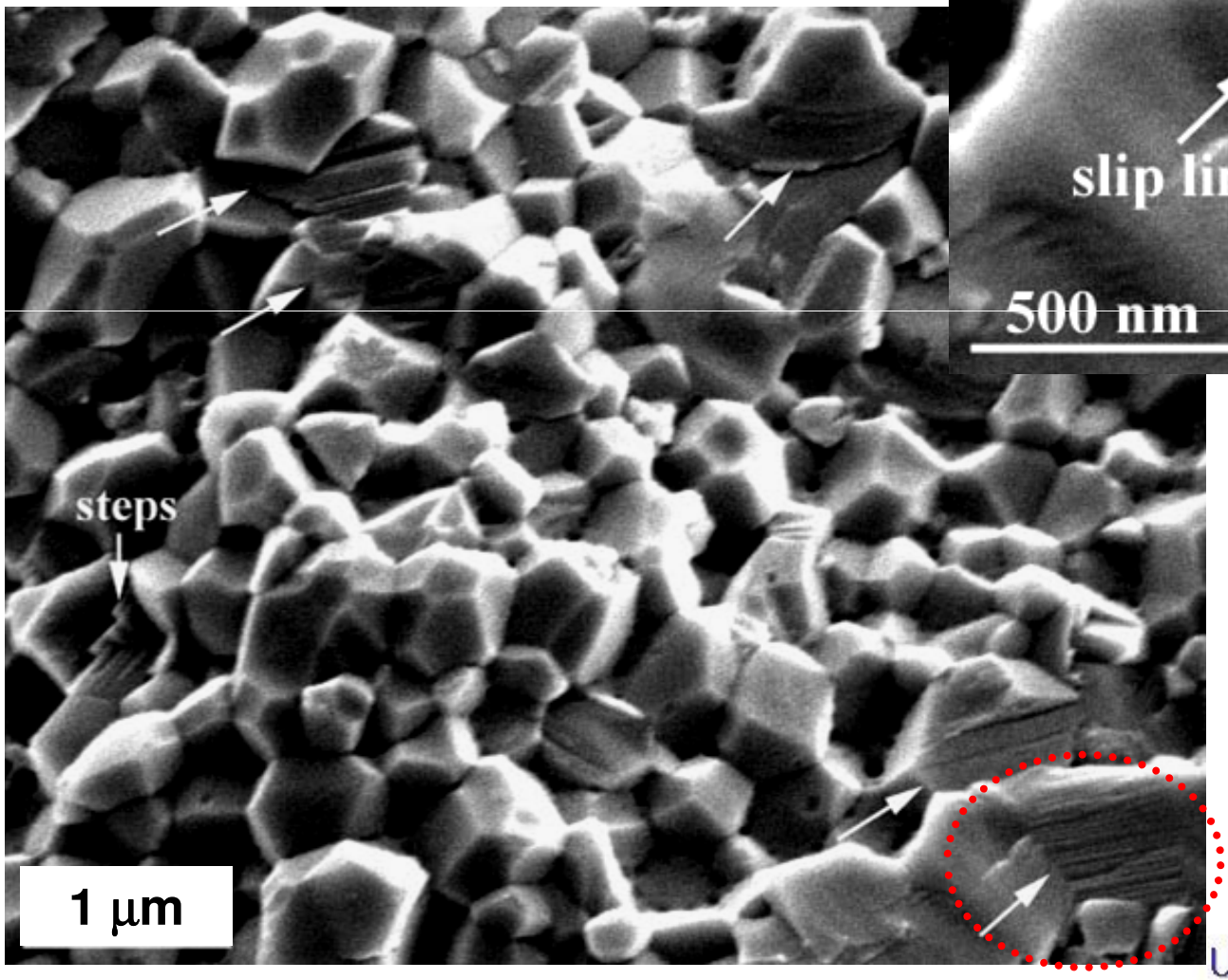

**Mixed Fracture (transgranular cleavage fracture + intergranular fracture)**

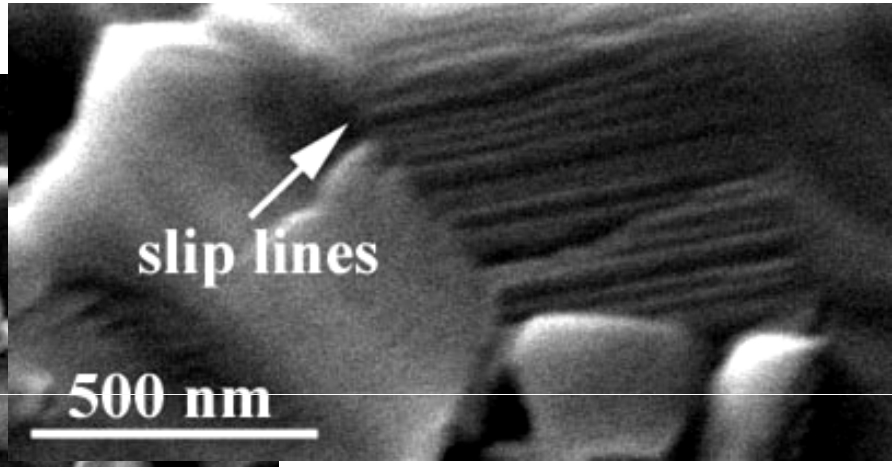



**Intergranular Fracture**

- **Average Grain Size -- 363 40 nm**
- **Strength -- 434MPa**

# Results and Discussion

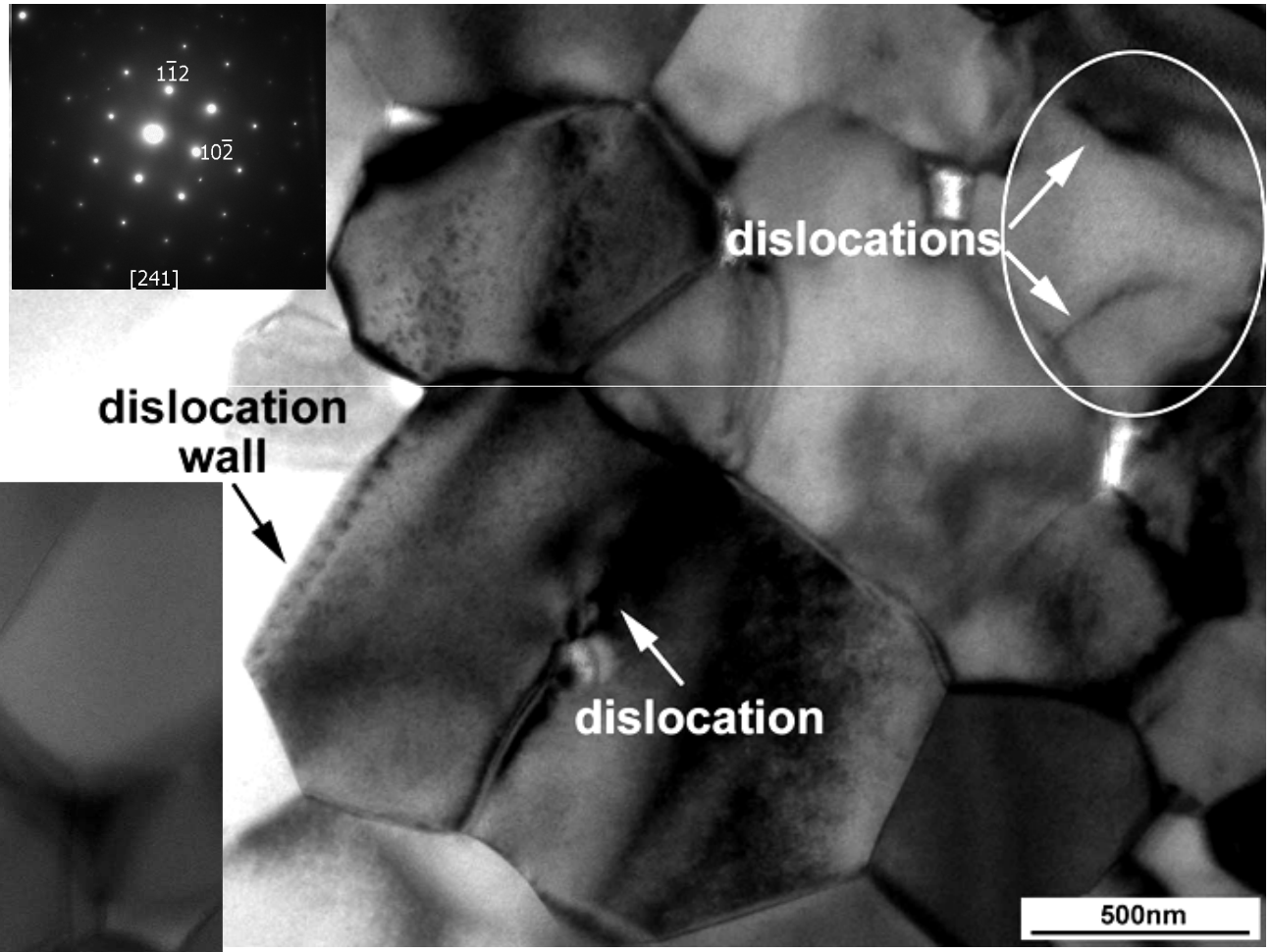


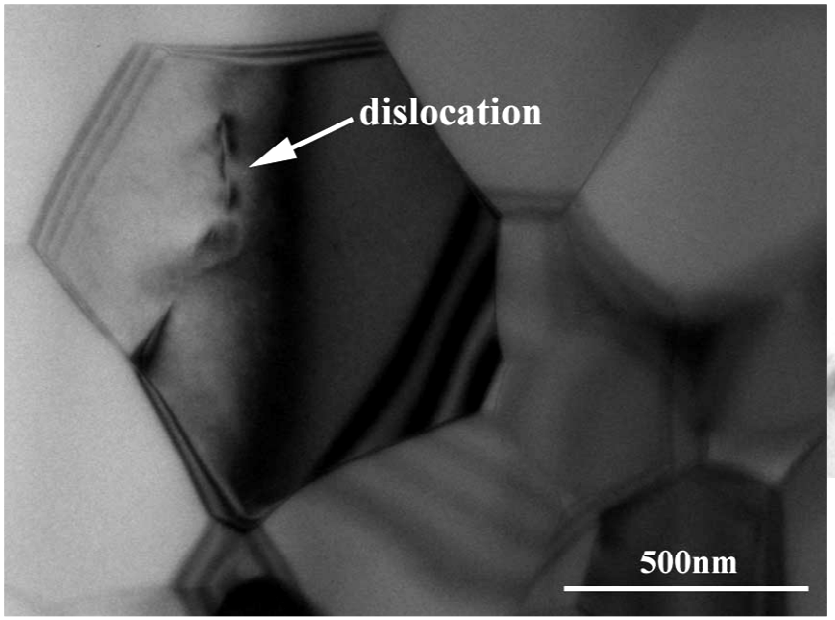




- **Average Grain Size -- 363 40 nm**
- **Relative Density -- 99.1%**
- **Strength -- 434MPa**

# Results and Discussion

- **Average Grain Size -- 363 40 nm**
- **Relative Density -- 99.1%**
- **Strength -- 434MPa**

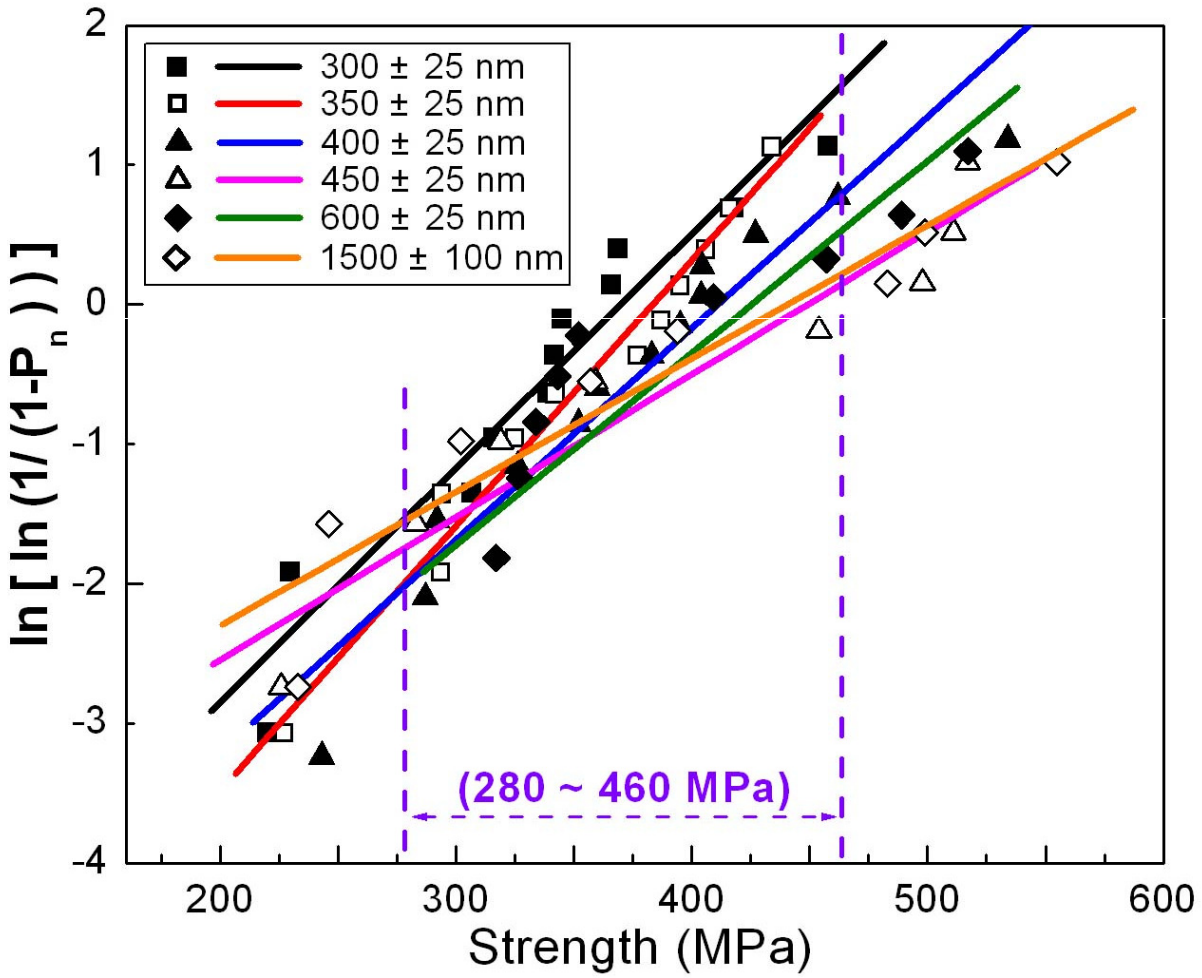


# Results and Discussion

- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness

# Results and Discussion



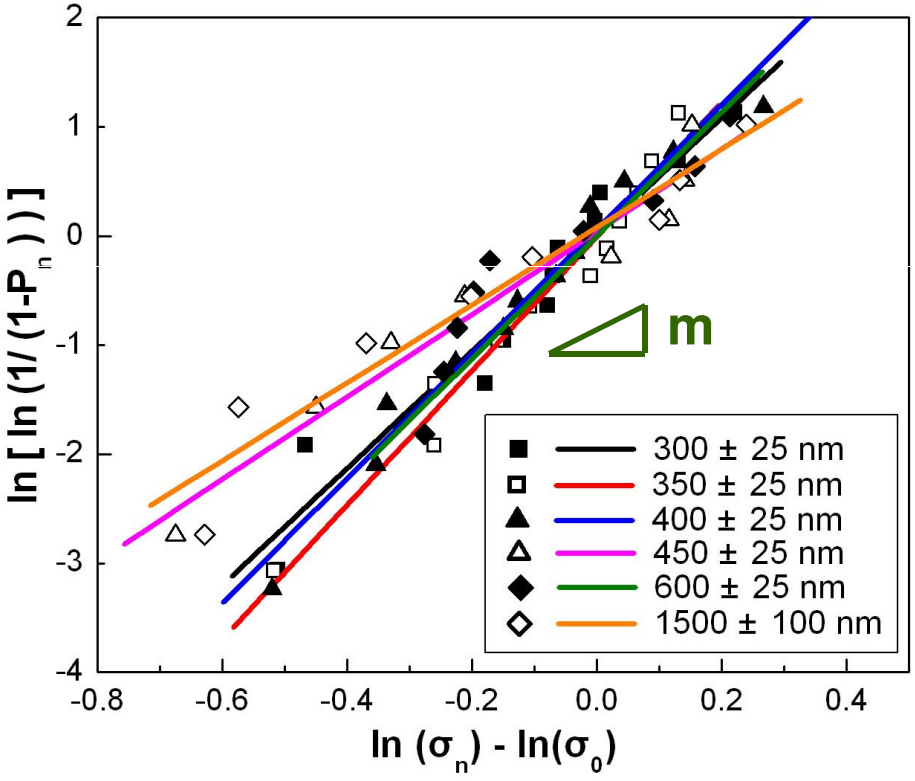

**Samples**

| Average Grain Size (nm) | Number of Samples |
|---|---|
| 300 25 | 11 |
| 350 25 | 11 |
| 400 25 | 13 |
| 450 25 | 8 |
| 600 25 | 10 |
| 1500 100 | 8 |

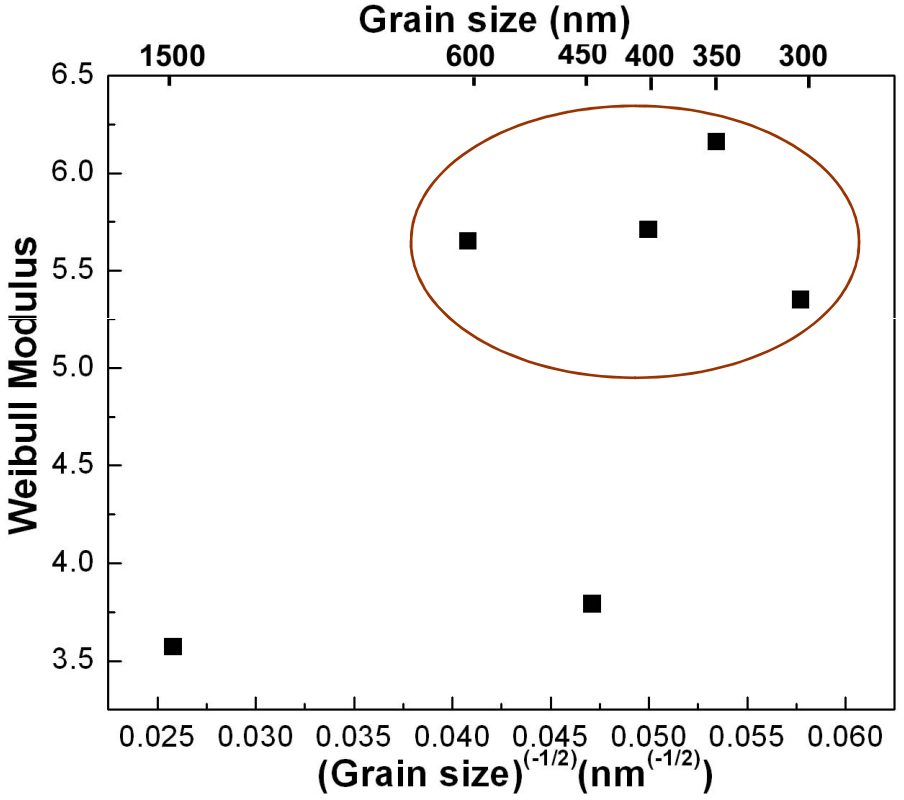
$$\ln[\ln(\frac{1}{1-P_n})] = m\ln\sigma_n - m\ln\sigma_0$$

# Results and Discussion

**m – Weibull modulus**

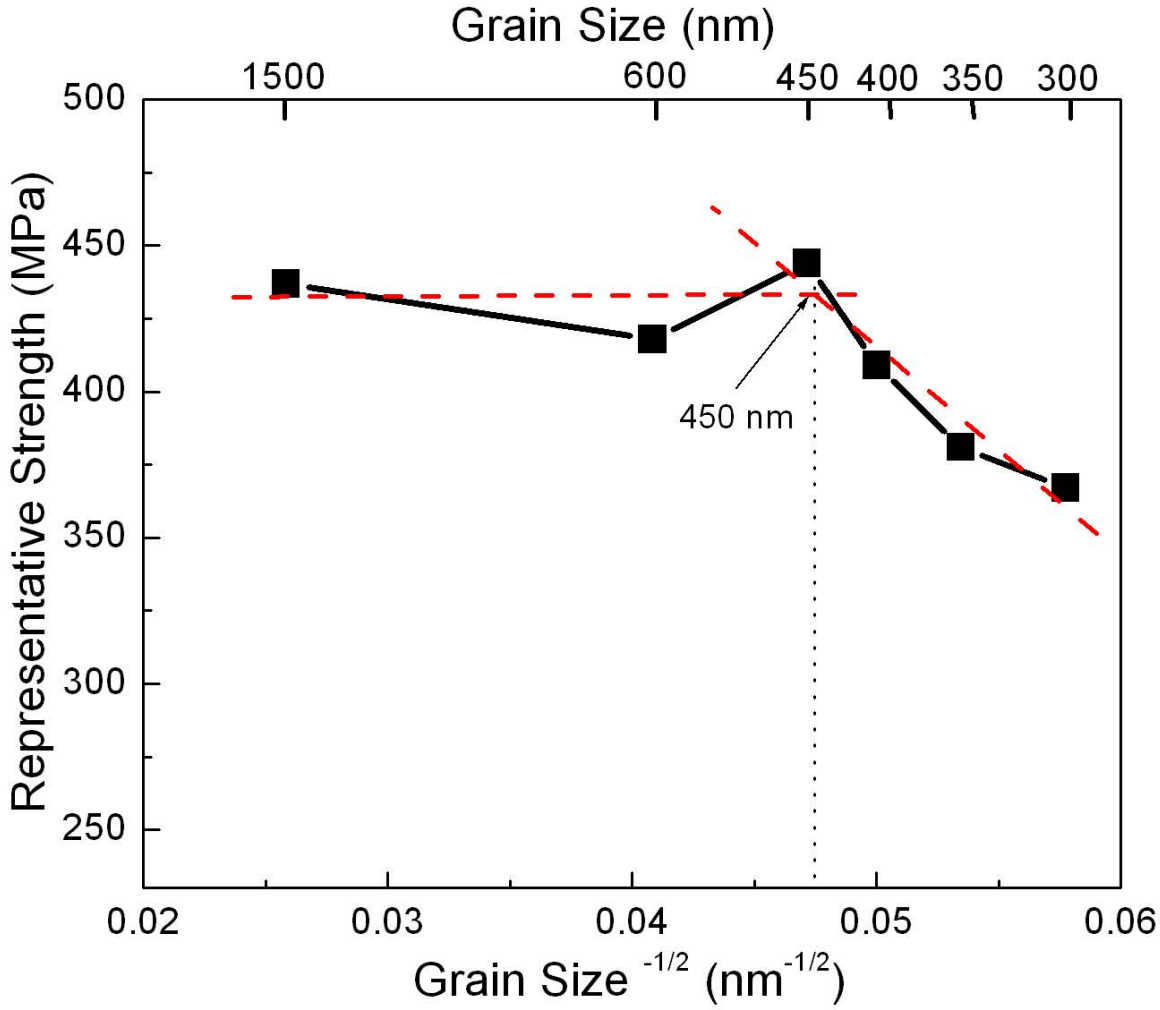



$$\ln[\ln(\frac{1}{1-P_n})] = m\ln\sigma_n - m\ln\sigma_0$$




**Typical Value: 5 - 20 for technical ceramics**

# Results and Discussion



# Results and Discussion

- Bending Process
- Fracture Surface Morphology
- Statistical Analysis
- Fracture Toughness

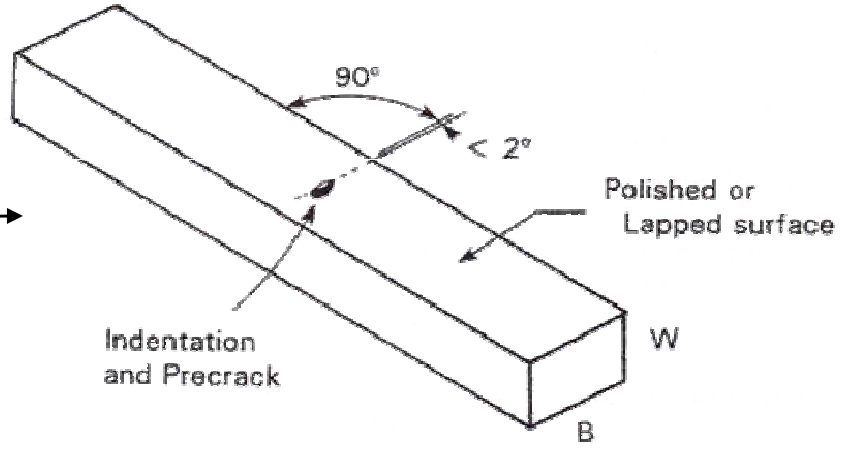
# Determination of Fracture Toughness of Ultrafine Alumina

- Vickers indentation

  Simple. SEM is used to measure the length

- Surface Crack in Flexure →

  ASTM C1421-01b, 2007



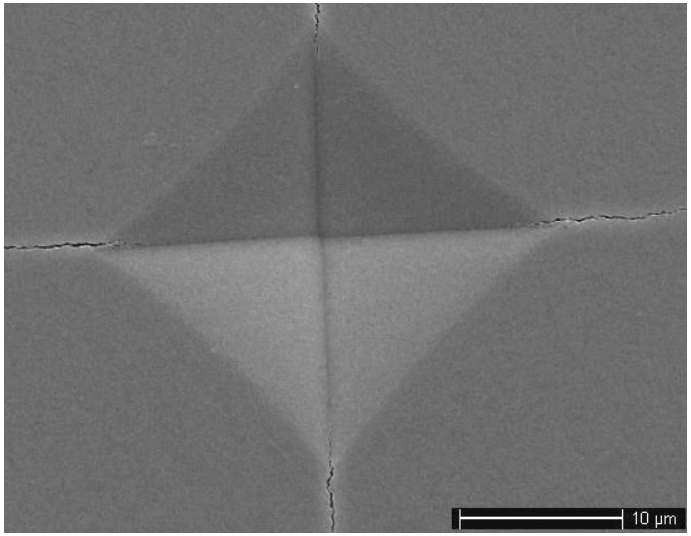

- Single-edge-V-notched beam (SEVNB)

  ASTM STP 1409, 2002

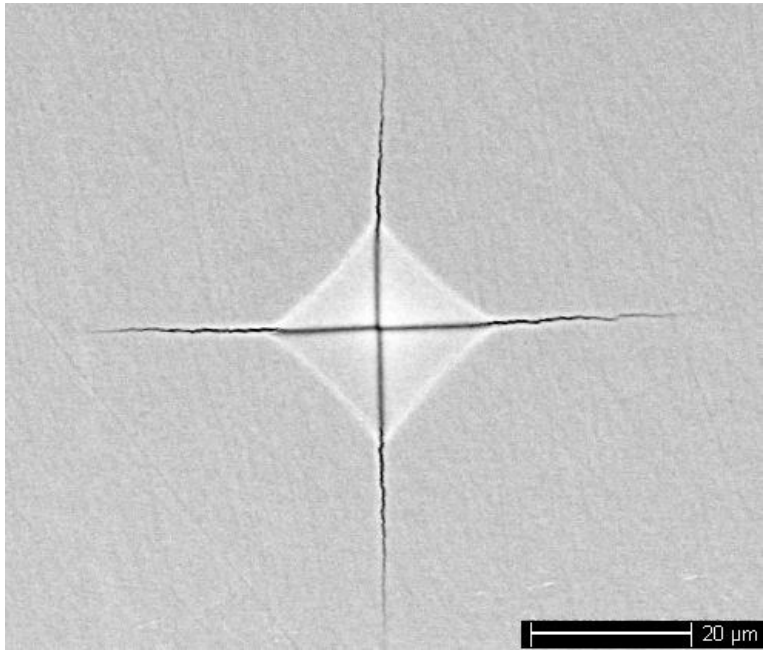
* It didn't give consistent results and was abandoned in our lab.

# Fracture Toughness determined by Vickers Indentation

- Surface of alumina was polished to 1μm finish before 10 indentations being made




Secondary electron image




Back scattering electron image

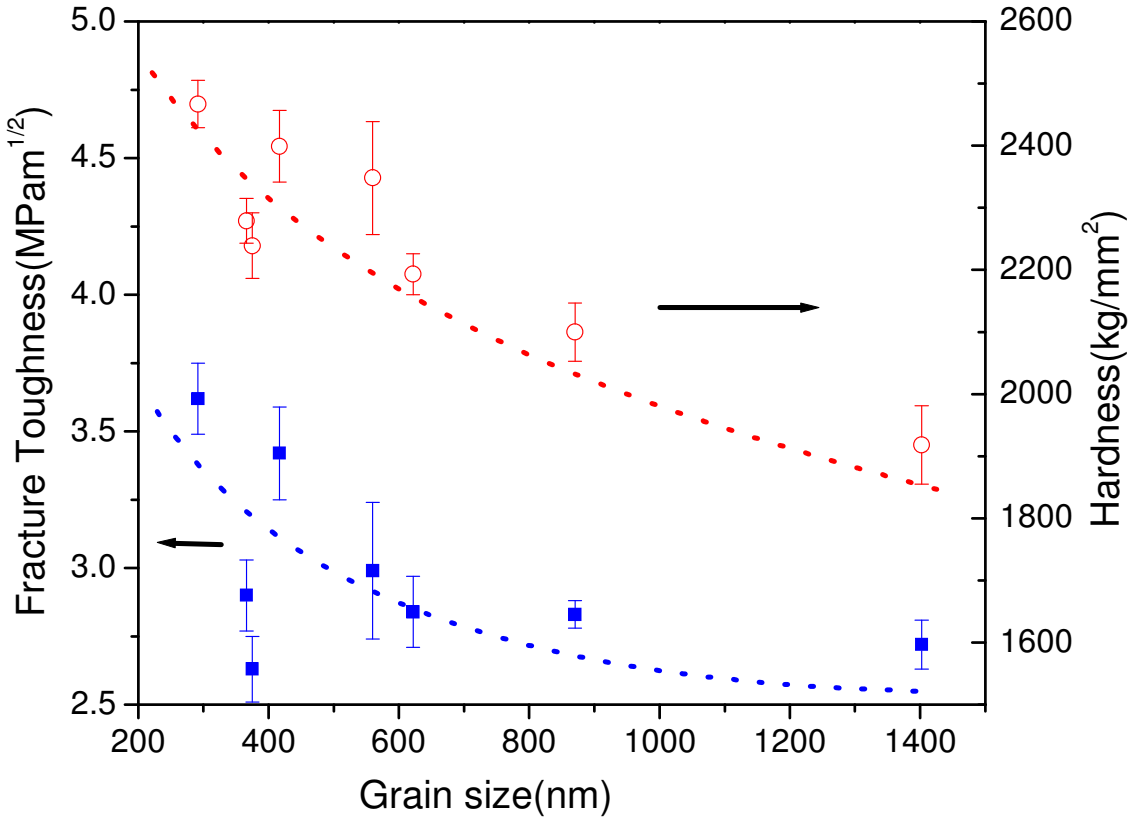
Anstis equation $K_{IC} = 0.016(E/H)^{1/2} P/c^{3/2}$ $H = 1.8544(P/d^2)$

H: Hardness E: Young's modulus P: Load d: length of diagonal of indentation
2c: length of diagonal of indentation and radial crack



G.R. Anstis et al., *J. Am. Ceram. Soc.*, 1980, 64(9), 533-538

# Fracture Toughness and Hardness from Vickers Indentation

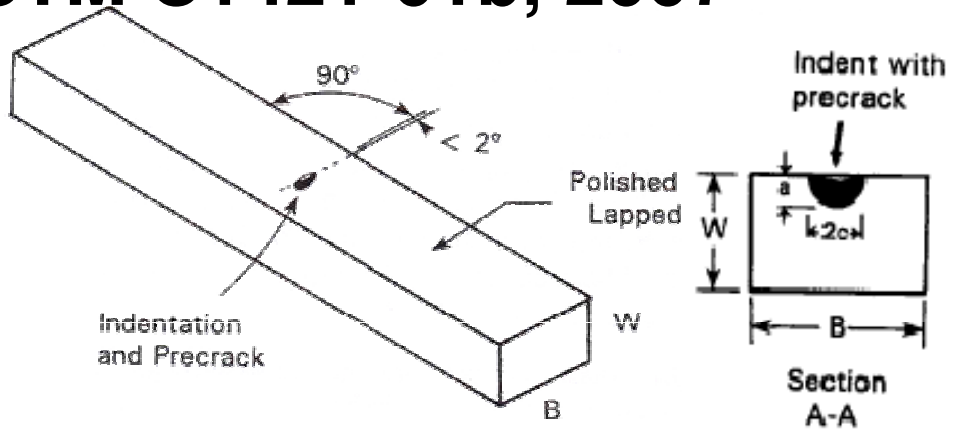



Dependence of fracture toughness and hardness of alumina on grain size

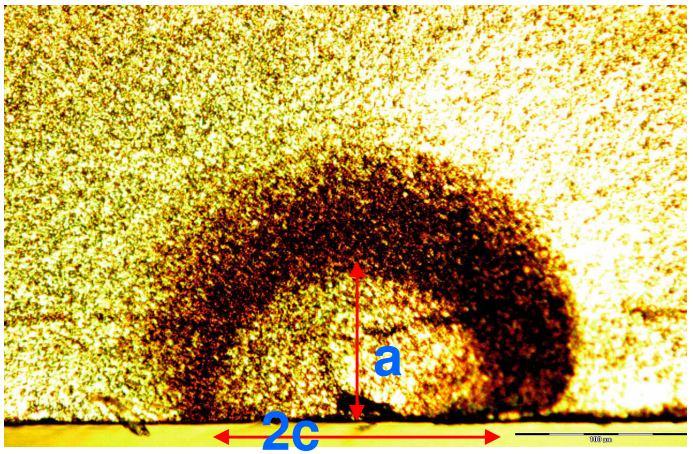
# Surface Crack in Flexure

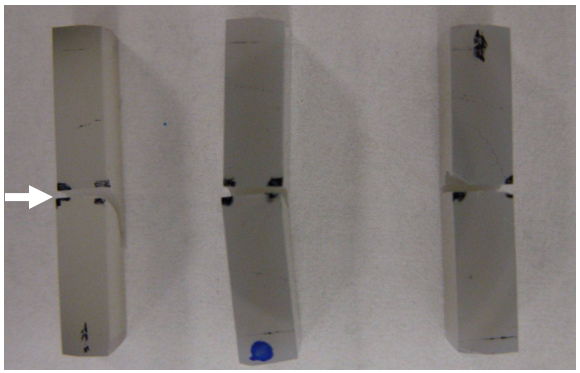
(Determination of Fracture Toughness of Advanced Ceramics)

## ASTM C1421-01b, 2007




Surface-Crack in
Flexure Test Specimen



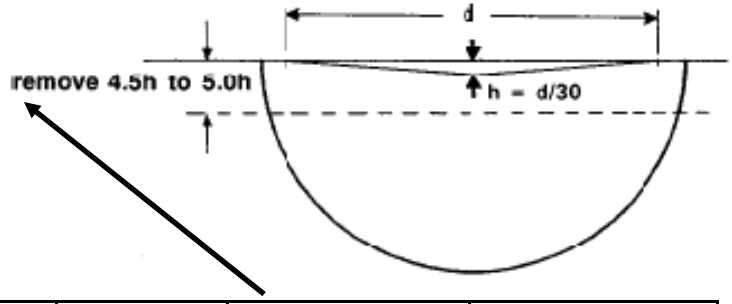



Knoop indenter precrack in a sintered alumina as photographed in optical microscope. The detect and measurement of precrack shape of width *2c* and depth *a* can be done in SEM or optical microscope.

# Surface Crack in Flexure

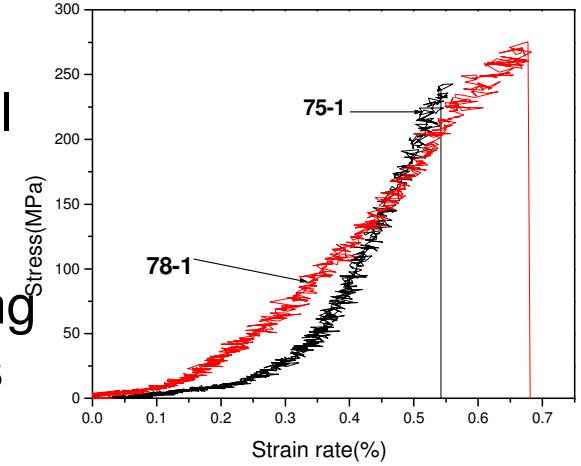
$$K_{IC} = Y\sigma\sqrt{a}$$

*Y*: stress intensity factor, *f( a, c, W)*
σ: flexure strength at fracture
*a*: precrack depth
c: half width of precrack shape
W: thickness of test specimen




**Flexure strength is most important to determine $K_{IC}$**

Typical four point bending curves




| Sample ID | Grain size (nm) | Flexure Stress (MPa) | Remove indenter and Damage zone? | Fracture Toughness ($MPa{\cdot}m^{1/2}$) (Surface crack in flexure) |
|---|---|---|---|---|
| 75-1 | 366 28 | 243 | Yes | 2.61 |
| 78-1 | 622 58 | 275 | Yes | 3.11 |
| 78-2 | 622 58 | 238 | No | 2.78 |
| 81-1 | 870±77 | 224 | No | 2.68 |
| 92-2 | 1402 249 | 257 | No | 3.02 |

# Comparison of Fracture Toughness of Alumina determined by Surface Crack in Flexure and Vickers Indentation

| Sample ID | Grain size (nm) | Fracture toughness ($MPa \cdot m^{1/2}$) (Surface crack in flexure) | Fracture toughness ($MPa \cdot m^{1/2}$) (Vickers indentation) |
|---|---|---|---|
| 75-1 | 366 28 | 2.61* | 2.90 0.13 |
| 78-1 | 622 58 | 3.11* | 2.84 0.13 |
| 78-2 | 622 58 | 2.78 | 2.84 0.13 |
| 81-1 | 870±77 | 2.68 | 2.83 0.05 |
| 92-2 | 1402 249 | 3.02 | 2.72 0.09 |

Single-Edge-V-Notched Beam (SEVNB):
$K_{IC}$ varies from 3 to 5 $MPa \cdot m^{1/2}$

Surface Crack in Flexure and Vickers indentation gave more consistent fracture toughness value in our lab.

* Removing indenter and damage zone

# Conclusions

- For dense ultra-fine grained alumina, most strength values fall within the interval of 280-460 MPa. The proper selection of appropriate sample preparation methods and preloading conditions is important to achieve accurate measurements.
- No dependence of Weibull modulus on grain size can be observed.
- Cleavage, transgranular and intergranular fracture as well as microcracks, slip lines and dislocations can be observed at or near the fracture surface.
- Surface crack in flexure and Vickers indentation gives more consistent fracture toughness values than does single-edge-V-notched beam (SEVNB).

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- ***Collaborators/Contributors***

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Thank You
for
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